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SUBJECT: Report of Industrial Waste Survey, Rocky Mountain Arsenal,
Denver, Colorado

THRU: The Surgeon General
ATTN: MCDPE-PO
Department of the Army
Washington, D. C. 20315

TO: Commanding General
US Army Materiel Command
ATTN: AMCPT-H
Washington, D. C. 20315

Inclosed herewith are nine copies of the report of subject study, performed by Captain LeRoy H. Reuter, MSC and 1st Lieutenant William F. Kilbury, MSC, this Agency, during the period 9-21 February 1965.

1 Incl
Rept of Proj No.
[illegible]

RICHARD J. PHILLIPS
Lt Colonel, MC
Commanding

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REPORT OF INDUSTRIAL WASTE SURVEY, PROJECT NUMBER 3471E19-59/66, ROCKY MOUNTAIN ARSENAL, DENVER, COLORADO

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THIS SURVEY WAS CONDUCTED TO DETERMINE THE PHYSICAL AND CHEMICAL CHARACTERISTICS OF LIQUID WASTES ENTERING THE INDUSTRIAL WASTE DISPOSAL SYSTEM AT RMA FOR A 12 DAY PERIOD. THE SURVEY WAS ALSO PERFORMED TO EVALUATE THE EFFECTIVENESS OF TREATMENT OF THE INDUSTRIAL WASTE AND TO DOCUMENT THE CHEMICAL NATURE OF THE WASTE INJECTED INTO THE DEEP WELL DISPOSAL SYSTEM.

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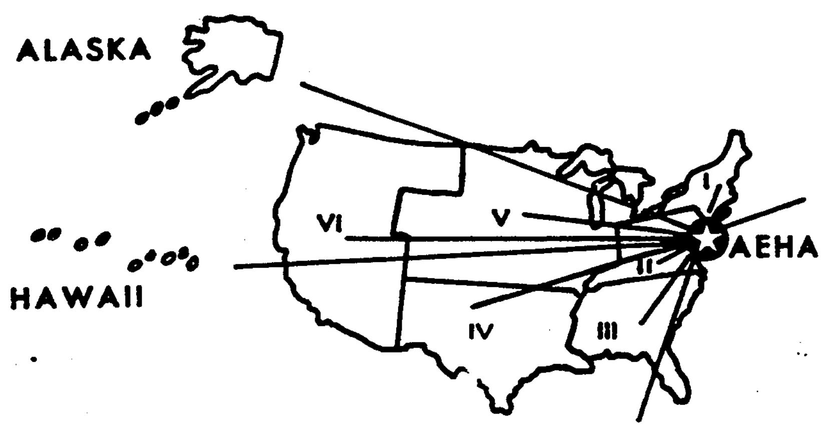
CLASS II ACTIVITY OF THE SURGEON GENERAL



SERVING THE ARMY IN ITS PREVENTIVE MEDICINE PROGRAM

REPORT OF INDUSTRIAL WASTE SURVEY
PROJECT NO. 3471E19-59/66
ROCKY MOUNTAIN ARSENAL
DENVER, COLORADO
9-21 FEBRUARY 1965

HEADQUARTERS
U S ARMY ENVIRONMENTAL HYGIENE AGENCY
UNITED STATES ARMY MEDICAL SERVICE

CLASS II ACTIVITY
OF
THE SURGEON GENERAL

EDGEWOOD ARSENAL
MARYLAND 21010

USAEHA-EE

22 SEP 1965

REPORT OF INDUSTRIAL WASTE SURVEY
PROJECT NO. 3471E19-59/66
ROCKY MOUNTAIN ARSENAL
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USAEHA-EE Rept of Ind Waste Sv, Proj No 3471E19-59/66, Rocky Mt Ars, Denver, Colo (9-21 Feb 65)

ABSTRACT

Sanitary Engineering Division personnel conducted the survey to determine the physical and chemical characteristics of liquid wastes entering the industrial waste disposal system at Rocky Mountain Arsenal for a twelve-day period. The survey was also performed to evaluate the effectiveness of treatment of the industrial waste and to document the chemical nature of the waste injected into the deep well disposal system.

The average flow of liquid wastes into the industrial waste system was 266.6 gallons per minute. Less than one sixth of the waste flow was injected down the deep well and the remainder cumulated to the existing volume of Lake F. This average influent flow was more than double that recorded in a survey conducted in 1960 by this Agency. A comparison of chemical properties indicate that the current waste is much more dilute than that of 1960. The chemical results also showed the current inlet stream exhibits wide variations in properties which are greatly stabilized by the treatment system, which consists of a one million gallon sedimentation lake and pressure filtration. Pesticide analyses indicated significant concentrations throughout the system, with the current treatment methods reducing the incoming pesticides concentration by one-half before deep well injections.

The disposal of all currently produced industrial wastes through the deep well system or some other adequate treatment system is an essential step in the elimination of Lake F. Greater effectiveness of the sedimentation lake can be obtained by relocation of the inlet and elimination of stagnant zones.

USAEHA-EE Rept of Ind Waste Sv, Proj No 3471E19-59/66, Rocky Mt Ars, Denver, Colo (9-21 Feb 65)

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REPORT OF INDUSTRIAL WASTE SURVEY
PROJECT NO. 3471E19-59/66
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DENVER, COLORADO
9-21 FEBRUARY 1965

1. AUTHORITY.

a. Para 4-6b(3)(d), AR 40-5, dated 10 April 1964.

b. Para 1-36b, Section VIII, AR 40-4, dated 15 May 1964.

c. Letter, SMUEA-EIS-C-CP, Headquarters, US Army Edgewood Arsenal, dated 2 July 1964, subject: Request for Assistance of the US Army Environmental Hygiene Agency (USAEHA), to this Agency, and indorsement thereto.

2. REFERENCES.

a. Preliminary USAEHA report, "Sanitary Engineering Project No. 3471E19-60/61, Industrial Waste Study, Rocky Mountain Arsenal, Denver, Colorado, 3-22 June 1960".

b. US Army Corps of Engineers, Omaho District, "Report of Ground Water Contamination, Rocky Mountain Arsenal, Denver, Colorado", September 1955.

c. US Army Engineer District, Omaho, "Preliminary Study Ground Water Contamination", Rocky Mountain Arsenal, 11 May 1960.

d. US Army Chemical Corps, "Staff Study Ground Water Contamination, Rocky Mountain Arsenal, 18 February 1960".

e. Preliminary USAEHA Report of Industrial Waste Study, Project No. 3471E19-59/65, Rocky Mountain Arsenal, Denver, Colorado, 9-21 February 1965.

3. OBJECTIVES. The objectives of this study were to:

a. Determine the quantities and characteristics of the waterborne industrial waste generated by all activities at Rocky Mountain Arsenal.

b. Evaluate the existing waste disposal facilities.

4. BACKGROUND.

a. General Background. Rocky Mountain Arsenal is located in the northeast section of Denver, Colorado. Original construction was completed in 1943 and the facilities were operated at full production during World War II producing lewesite, mustard, arsenous chloride, and chlorine gas and filling munitions with white phosphorus. Industrial wastes were stored in a number of clay bottom evaporation lakes. The east plant facilities were leased in 1947 by Julius Hyman Company and used for the production of insecticides. In 1952, the Shell Chemical Company took over the operation of the insecticide facilities. The GB Plant was constructed during 1952 and production continued from 1953 to 1956. Filling of munitions with CW-agents, the demiliterization of mustard-filled munitions and the blending of hydrazine have been the principle military operations contributing industrial waste since 1956. Figure 1 shows the physical arrangements of Rocky Mountain Arsenal industrial facilities.

b. Crop damage in the area adjacent to and northeast of Rocky Mountain Arsenal was first noticed in 1951 and the damage was associated with the use of the shallow well irrigation water. The US Corps of Engineers concluded in a 1955 report that the Arsenal waste lakes were the main source of contamination of these shallow wells.

c. In a report by the Ralph M. Parsons Engineering Company in September 1955 it was recommended that sealed evaporation-storage lakes be provided and an acid treatment plant be built. Acid and salt were to be reclaimed and sold, and the remaining waste was to be retained and evaporated in the sealed lakes.

d. Waste disposal remained unchanged, however, until October 1955 when complete retention of the wastes produced was initiated to eliminate additional ground water contamination. Lake F, the largest of the evaporation-storage lakes (96 acres and approximately 200 million gallons at full capacity) was sealed with an asphalt membrane. For additional protection, the membrane was covered with approximately 12" of compacted soil. Sealing Lake F was only an interim measure since the continuing waste flow exceeded the anticipated losses through solar evaporation.

e. In March 1960, A. J. Ryan and Associates, Incorporated, Consulting Engineers, concluded in a report, "Comparative Methods of Treatment of Waste Effluents at Rocky Mountain Arsenal, Denver, Colorado", that the most practical means of waste disposal would be evaporation.

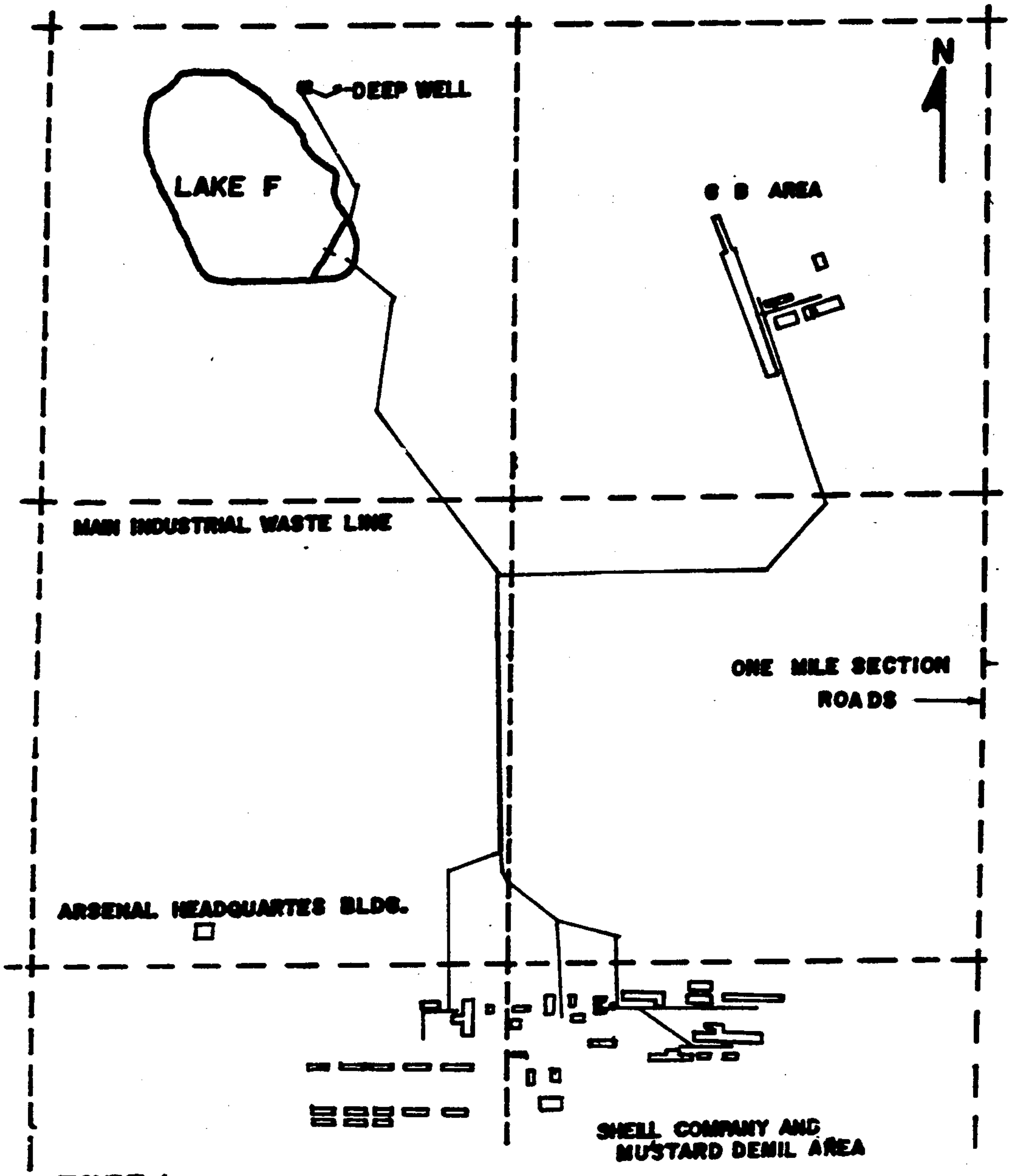


FIGURE I
INDUSTRIAL WASTE COLLECTION SYSTEM, ROCKY MT. ARSENAL

Deep well injection of the waste was concluded to be practicable if the wastes were injected in a low suspended solids condition. Dilution of the waste with surface water was concluded to be unacceptable, because of non-availability and water reuse policies.

f. This Agency performed an industrial waste survey in June 1960 to provide information on the sources, volume of flow, and composition of the waste for use in the evaluation of waste treatment methods (para 2a).

g. E. A. Polumbus, Jr., and Associates, working as Consulting Engineers for the US Army, concluded in a January 1960 report that a geological investigation indicated that with a reasonable degree of certainty, 800 gallons per minute (gpm) of waste water could be disposed of in a deep well drilled on Arsenal property. It was estimated that the cost of drilling the deep well to an 11,400' proposed depth, plus all necessary facilities for treating and injecting the waste at a rate of 800 gpm would be approximately $1,000,000. This recommended solution was ultimately accepted and a deep injection well designed to handle 800 gpm at a surface pressure of 2,500 pounds per square inch (psi) was drilled to a depth of 12,075 feet. Surface pretreatment facilities included a clarifier and asbestos-diatomaceous earth coated pressure filters designed to reduce suspended solids concentration to 20 milligrams per liter (mg/1) or less. The system was placed in operation early in 1962.

h. After a few months of operation, removal of suspended solids from the Lake F waste became very expensive as coagulation became more difficult and filter runs shorter. Most of the difficulties were attributed to an unfilterable bacterial slime. The use of the deep well was discontinued in mid-1963 because of these high pretreatment costs. To offset the increasing waste volume in Lake F, a spray evaporation raft was placed in operation. The combined evaporation rate from the surface of Lake F and the spray system reportedly average approximately 140 gpm during the eight months of the year when climatic conditions are most favorable for operation.

i. Plant Processes.

(1) <u>Shell Chemical Company Operations</u>. The Shell Chemical Company has manufactured the following pesticides at the Arsenal: Aldrin, azidrin, bidrin, ciodrin, dibrom, dieldrin, endrin, parathion, methyl parathion, vapona, phosdrin, nemagon C and Compound 4072. Azidrin, bidrin, ciodrin, phosdrin and Compound 4072 were not produced during this survey and Shell anticipates reducing the production of aldrin, dieldrin and endrin.

(a) Aldrin. Aldrin, produced in Building 422, is a highly chlorinated cyclic hydrocarbon produced by the Diels-Alder diene reaction. Production involves peroxide oxidation in agitated, jacketed kettles. Waste was primarily wash water combined with a $CaCl_2$ brine solution and organic solvents. The brine was derived from dryers at an estimated rate of 2 gpm. Additional wastes include diatomaceous earth from filters which is diluted with water and trucked out to Lake F for disposal.

(b) Dieldrin. Dieldrin, produced in Building 516, is an epoxide of aldrin formed by reacting peracetic acid with aldrin. Manufactured by a batch operation, the primary wastes were acetic acid, wash water and hydrogen peroxide. Estimated quantity of waste was 500 gallons per batch of highly concentrated acetic acid and 1 to 2 gpm from a continuous centrifuge.

(c) Endrin. Isodrin, an isomer of aldrin, is converted to endrin by reaction with peracetic acid in Buildings 515 and 516. Wastes included acetic acid, sodium chloride, suspended carbon, and calcium chloride from dryers.

(d) Methyl Parathion and Parathion. Production facilities for these products are located in Building 514. Both are made in batches from p-nitro sodium phenolate (PNSP) and dimethyl phosphoro chloro-oridothionate. The wastes which are discharged to the sewer after hydrolysis are p-nitro phenol and diethyl ortho-thiophosphonic acid plus excess caustic. The estimated waste quantity which requires hydrolization is about 1000 gallons per batch.

(e) Vapona, Nemagon C and Dibrom. All three products are manufactured in Building 471. Vapona is the addition product of chloral (Trichloro aldehyde) and trimethyl phosphite. Before the reaction chloral is treated in concentrated H_2SO_4 to remove polymers, producing an acid waste stream. The estimated waste flow is approximately 400 pounds of concentrated H_2SO_4 and 400 pounds of chlorinated hydrocarbon per batch of Vapona plus an additional dilute waste flow of about 50 gpm from steam jets. Dibrom is made by the addition of bromine to the unsaturated bond of Vapona. A steam jet with a 48 gpm discharge is used for this production. Nemagon C, a soil fumigant, is the addition product of bromine and allyl chloride, which produces 15 gpm dilute waste flow from a condensor jet. In addition to the above waste flows, this system also produces 60 gpm flow from the hot well dilution condensor. These condensor jets would not be expected to be in operation more than an average of 8 hours per day.

(2) Rocky Mountain Arsenal Operations. The industrial waste producing facilities of the Arsenal are located in all of the installation plants areas.

(a) Mustard Demiliterization. The demiling facilities are located in Building 538 of the east plants area. A waste stream is derived from cooling water used on the incinerated bombs. During the study period, the facilities were used to demiliterize 155 mm shells which were air dryed so that only a very dilute waste was contributed to the industrial waste sewer.

(b) GB Plant. The GB Plant facilities for manufacturing and filling of munitions are located as indicated in the general layout sketch (Figure 1). The manufacturing facilities were not in operation during the study period; however, GB was being redistilled during the survey and munitions were being filled with GB and Vx. Waste consisted primarily of the hydrolysis products of the CW agents, caustic solution and wash down. Spray paint activities are also included in Building 1601 in the GB Plant area; however, the water used for the screen in the booths is recirculated and waste flow is negligible. Total average waste flow from the GB plant area was approximately 3 gpm.

(c) Hydrazine Blending Plant. Hydrazine blending facilities are located in Building area 755. Waste is produced from scrubbers for vapors produced during the blending operation. Approximate average flow is 3 gpm. Waste flow is into a sump with gravity flow into the industrial waste line.

(d) Laundry. Laundry facilities located in Building 314 generate a waste composed of wash water, detergents and dyes. Average waste flow from the laundry during the survey was approximately 3000 gallons per day.

(e) Quality Surety Lab. Small quantities of laboratory wastes are generated from this activity which is located in Building 313. Control analyses are performed on GB and other CW agents which produce small quantities of waste which goes into the industrial sewer. Rocky Mountain Arsenal officials estimate this industrial waste flow to be 10,000 gallons per month.

j. Industrial Waste Disposal Facilities.

(1) General. All industrial waste water flows into Lake F-1, a "diked off" portion of Lake F. Shell's parathion wastes are hydrolyzed

before discharge into the sewer lines but all other wastes flow from their point of generation without treatment into a trunkline which flows approximately two miles by gravity to Lake F-1. Part of the waste from Lake F-1 overflows into Lake F while the remainder is discharged down the deep well. Waste discharged down the well flows from either Lake F or F-1 through a clarifier to a wet well, is pumped through pressure filters to a storage tank and then down the deep well. A dry chemical feeder and flocculator, although available, have not been in use as stated in para 4h. No waste from Lake F has been injected down the deep well since 1963.

(2) Shell Chemical Company's Waste Hydrolysis Tank. The caustic treatment procedure used for parathion wastes consists of maintaining a 140°F temperature for approximately 16 hours with a caustic supply in excess of at least 10 times the stoichiometric requirement. Mixing is achieved by injecting 20 percent NaOH into the suction side of a circulating pump which discharges into the hydrolysis tank.

(3) GB Plant Sump. All industrial wastes from the GB plant flow into a collecting sump equipped with a float-activated pump having a reported maximum capacity of approximately 200 gpm at the float activated head level which pumps waste into a sewer line leading to Lake F-1. Within the plant, detoxification tanks are provided in which rejected batches of agent may be dumped, and detoxified with Cl_2 and NaOH before being dumped into the contaminated sump.

(4) Lake F-1. Lake F-1, which has an approximate capacity of one million gallons, receives the total industrial waste flow. A parshall flume located on the effluent channel provides flow measurement. Lake F-1 is divided approximately into two equal volumes (see Figure 2) by an old dirt road which serves as a dike. The overflow, allowing waste to flow from F-1 to F, is located at the cannel neck connecting the two sections of the lake. Lake F-1 is lined with an asphalt membrane covered with approximately 12 inches of compacted soil.

(5) Dry Chemical Feeder and Flocculater. A dry chemical vibrating type feeder with rated capacity from 0 to 300 lbs/day is available but has not been used. The unit has a dissolving chamber, mechanical mixer, and 4½ gpm at 16' head injection pump.

(6) Clarifier. The circular unit is 38' in diameter and depth to the notched weir is 9 feet. The clarifier is equipped with a rake type sludge collector, influent diffuser, and surface scum skimmer. Influent is fed into the center of the unit. This EIMCO-Process Equipment was designed for an overflow rate of 500 gals/ft^2/day.

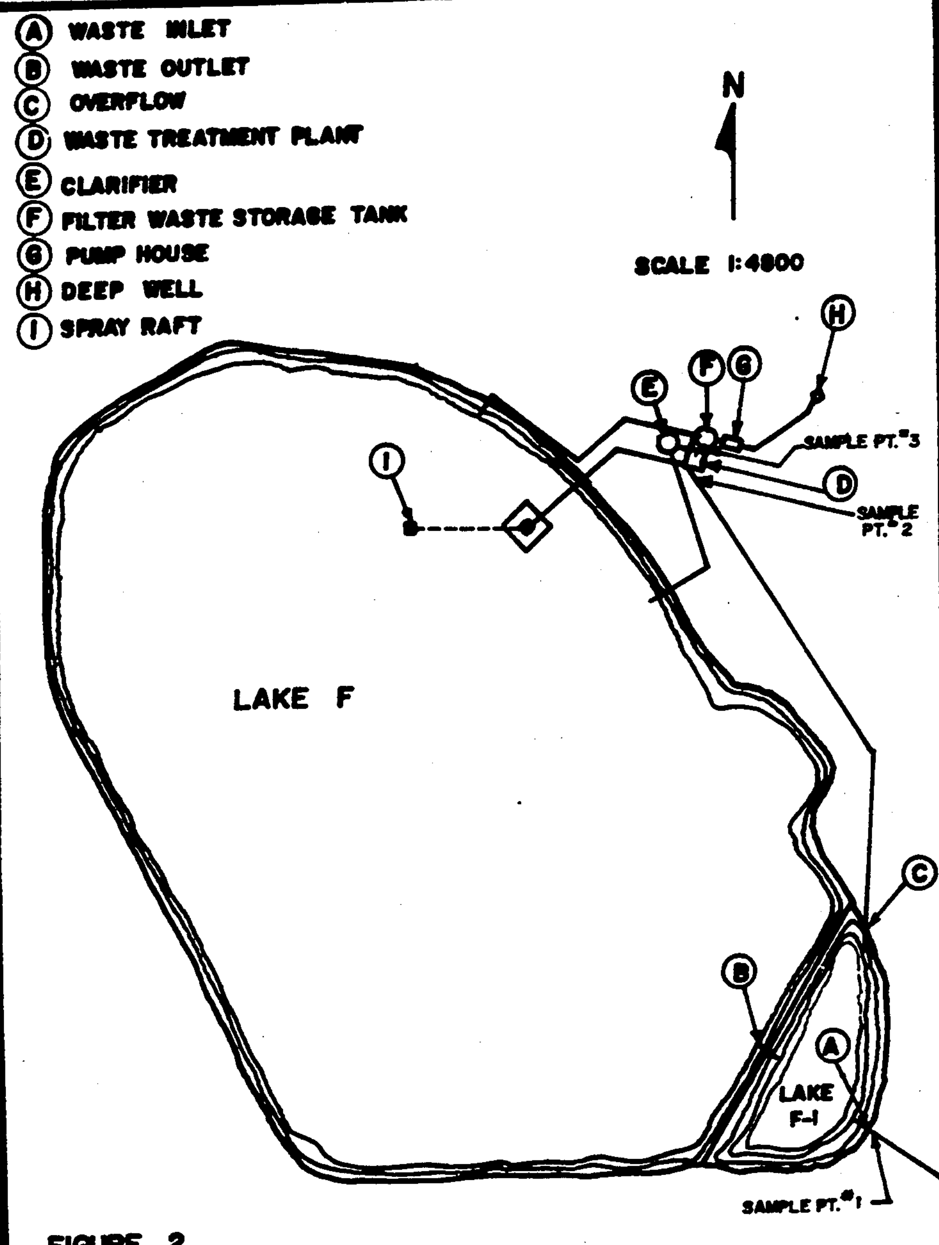


FIGURE 2

INDUSTRIAL WASTE LAKES, ROCKY MT. ARSENAL

(7) Filters. Two pressure filter units manufactured by United States Filter Company are operated in parallel. They are designed to remove suspended solids by means of an asbestos filter-diatomaceous filter coat from 400 gpm waste flow under 65 psi working pressure. The filters are cylindrical, multiple screen type with center discharge and bottom sludge removal. The filters are cylindrical, multiple screen type with center discharge and bottom sludge removal. The filter leaves are made up of five layers of screening. The screen area per filter is approximately 400 square feet and can be increased to 500 square feet. The filter leaves are cleaned by high pressure water jets.

(8) Storage Tank. Filtered waste is pumped to a 50,000 gallon capacity circular storage tank, 24' in diameter and 15' high.

(9) Deep Well. The deep well was designed to accommodate a waste volume of from 400 to 800 gpm at 2500 psi surface injection pressure. To effectively isolate the injection reservoir a 20" casing was set at 135 feet; a 13-3/8" casing at 2020 feet, and 8-5/8" casing at 11,171 feet and a 5½" liner placed from 1,007 to 11,975 feet. To further insure complete isolation, each string of casing was sheathed with cement for its entire length. The annual volume between the 5½" injection tubing and the 8-5/8" casing is filled with fresh water and a pressure sensing and recording device at the well head is activated if any leaks occur from the closed chamber. The geological formations and the depths at which they were encountered are:

Geological Formation	Depth to Top of Formation (ft)
Fox Hills	1,250
Pierre Shale	1,480
Hygiene Zone	5,448
Niobrara	7,710
Carlile Shale	8,078
Greenhorn Limestone	8,120
Granerous Shale	8,345
"J" Sandstone	8,485
Dakota Sandstone	8,633
Lakota Sandstone	8,730
Morrison Formation	8,786
Lykins Formation	8,972
Lyons Formation	9,582
Fountain Formation	9,772
Regolith	11,880
Ordovician Cambrian	11,895
Pre-Cambrian Schist	11,950
Pre-Cambrian Gneiss	11,970

Although not in use during the survey, four .95 gpm TDH 4650' reciprocating Multiplex plunger pumps are available to inject waste down the deep well.

5. PROCEDURES. To obtain the required data, the US Army Environmental Hygiene Agency sent out a survey party which included two sanitary engineers and four enlisted technicians.

a. Sample Collection. The sample period for the survey extended from 0700 hours, 9 February 1965, to 0700 hours, 20 February 1965. The sample day ran from 0700 hours one day to 0700 hours the following day.

(1) Sample Point Locations. The locations of the sample points are shown in Figure 1.

Sample Point 1 - Influent to Lake F-1
Sample Point 2 - Effluent from Lake F-1
Sample Point 3 - Effluent from pressure filters

(2) Grab Samples.

(a) Grab samples, 1000 ml volume, were taken at points 2 and 3 every two hours.

(b) Flows at points 2 and 3 were measured at time of sampling from readings on the flow meter at the filters. Since the pressure and head on the filters were relatively constant, flow was considered uniform throughout any two hour period. The flow meter had been calibrated before the survey.

(3) Composite Samples.

(a) An automatic sampler was installed at point 1 and adjusted to collect a sample every 15 minutes. The sample volume from eight consecutive 15-minute interval periods was collected at each odd numbered hour of the sampling day and was retained. At the end of the sample day the two-hour samples were composited in proportion to flow at point 1. Portions of each composited daily sample were returned to USAEHA for additional analyses.

(b) Flows at point 1 were measured by an existing Parshall Flume equipped with an automatic recording device.

(c) Composite samples for points 2 and 3 were made by proportioning 350 ml volumes from each grab sample at these points. Portions of each daily composited sample were returned to USAEHA for additional analyses.

(4) Special Samples and Studies.

(a) Waste samples from the aldrin process still bottom were collected and returned to USAEHA for analysis.

(b) Samples from the ice cover on Lake F and Lake F-1 were collected and analyzed.

(c) Samples of bottom sludge from Lake F and F-1 were collected and returned to USAEHA for analysis.

(d) Tracer Studies. Congo red waste from the Rocky Mountain Arsenal laundry and a commercial fabric dye were used as tracers in determining the flow pattern in Lake F-1. In addition to visual observations, samples were manually collected periodically at five locations and analyzed for tracer concentration.

b. Methods of Analysis.

(1) Laboratory facilities in the treatment plant and in a section of the Quality Surety Laboratory at Rocky Mountain Arsenal were utilized. Where noted, certain samples were returned and analyses completed at USAEHA and USA Chemical Research and Development Laboratories (USACRDL).

(2) Grab Samples. Analyses were in accordance with the schedule shown in Table 1. The temperature readings were made in situ, while pH, specific conductance, acidity/alkalinity, and turbidity were measured in the treatment plant laboratory. Turbidity measurements were made with the Hellige turbidimeter calibrated with a standard SiO_2 solution.

(3) Composite Samples. Analyses were in accordance with the schedule shown in Table 1. All determinations were those as described in Standard Methods for the Examination of Water and Wastewater, Eleventh Edition, 1960, with the following exceptions:

(a) Fluoride - "Ion-Exchange Method for Determination of Fluorides in Potable Waters", Kelso, Toymae S., Mathews, John M. and Kramer, Harry P., Analytical Chemistry, Vol 36, 1964, p 577. This method was used because of its simplicity, reproducibility and freedom from interference

(b) COD - "Elimination of Chloride Interference in the Chemical Oxygen Demand Test", Dobbs, R. A., and Williams, R. T., Analytical Chemistry, Vol 35, 1963, p 1064. This method was used to avoid high salt concentration interferences.

TABLE 1

SCHEDULE FOR ANALYSES

ANALYSES	SAMPLE POINT NUMBER 1	2	3
Two-Hour Interval Samples			
1. Acidity/Alkalinity	2H	2H	2H
2. pH	2H	2H	2H
3. Specific Conductivity	2H	2H	2H
4. Temperature	2H	2H	2H
5. Turbidity	2H	2H	2H
24-Hour Composite Samples			
1. Acidity/Alkalinity	D	D	D
2. Aluminum	D	D	D
3. Ammonia Nitrogen	D	D	D
4. Arsenic	D	D	D
5. Calcium	D	D	D
6. Chemical Oxygen Demand	D	D	D
7. Chloride	D	D	D
8. Fluorides	D	D	D
9. Insecticides	D	D	D
10. Iron	D	D	D
11. Kjeldahl Nitrogen	D	D	D
12. Magnesium	D	D	D
13. pH	D	D	D
14. Phenols	D	D	D
15. Solids: Dissolved	D	D	D
Volatile	D	D	D
Volatile Settleable	D	D	D
Total	D	D	D
Total Volatile	D	D	D
16. Specific Conductance	D	D	D
17. Sulfates	D	D	D
18. Sulfides	D	D	D
19. Total Phosphorus	D	D	D
20. Turbidity	D	D	D

2H - Every two hours D - Daily

(c) Iron - The standard tripyridine method was not reproducible because of the high phosphate concentration of the waste. The actual procedure used is described in Appendix I.

(d) Arsenic - The Robert A. Taft Sanitary Engineering Center, Cincinnati, Ohio, silver diethyldithiocarbamate method was used to provide a limit of sensitivity low enough for the anticipated concentrations. The procedure used is described in Appendix I.

(e) Sulfide - "Sulfur by Evaluation as Hydrogen Sulfide", Snell, F. D., and Snell, C. T., Colorimetric Methods of Analysis, Vol 1, Sixth Printing, 1945, p 598. This method was used because of its simplicity and the interferences to other applicable procedures.

(f) Phosphorus - The phosphorus method is described in Appendix I and was used because it is less sensitive to interferences than the method in Standard Methods for the Examination of Water and Wastewater.

(4) Special Analyses.

(a) Pesticide Analyses. Gas chromatography was used to measure the presence of the following pesticides: Aldrin, dieldrin, endrin, methyl parathion, parathion, nemagon C and vapona. These determinations were performed on selected daily composite samples from sample points 1, 2 and 3 and mud samples taken from the bottom of Lake F-1. Complete instrumental data for the gas chromatography unit used for these determinations is given in Appendix I. Each liquid sample was pretreated by extraction with spectrograde hexane in a liquid-liquid apparatus as described in Analytical Chemistry, Vol 36, pp 1340-3, June 1964. A one liter waste sample was extracted two times with 250 ml amounts of hexane for 3½ hours each. The two extractions were combined, dried with 2 gm of K_2SO_4 and concentrated to 30 ml by distillation prior to analysis by the gas chromatograph. Mud samples were extracted by taking 50 ml of the sample, diluting to 1 liter with distilled water and adding 100 ml of hexane. The two liquid layers were mixed together with a magnetic stirrer for one hour and then the hexane layer decanted. This procedure was repeated and the two hexane extracts were combined and then dried and concentrated in the same manner as in the pretreatment of the liquid waste samples.

(b) Spectrographic Analyses. Spectrographic methods were used for the analysis of calcium, aluminum and magnesium. Spectrographic plates were run in the 2300-3400 Angstrom range in duplicate for

each specimen. Calcium was evaluated by flame spectrophotometry to serve as an internal standard. Aluminum and magnesium were read on the film plates and negative log ratios were set up as absorbance ratios. The ratios were then multiplied by plate factors and by the value taken for the calcium standard. Plate factors were found by running six spectra of mixtures of calcium, aluminum and magnesium salts at various volumes to determine relative concentration responses on the electrodes, and then averaging these responses to obtain factors for calcium:aluminum and calcium:magnesium ratios.

6. FINDINGS.

a. Production Activities.

(1) Shell Chemical Company Pesticide Production. A list of the productions activities of Shell during the survey is shown in Table 2. For each sample day the amount of pesticide made is listed by number of batches and, as indicated, the production runs of certain pesticides were discontinued during the survey. For the two weeks of the survey, it is estimated that the Shell Company building complex consumed a total of 94,000,000 gallons of water which is an average of 4630 gpm. (This is about 97 percent of water consumed on the Arsenal.) This figure includes all water uses such as steam production, cooling, and other process water.

(2) Other RMA Industrial Waste Producing Activities. During the period of 8-21 February 1965, the following approximate waste flow estimates have been provided by Rocky Mountain personnel. These estimates are based on a five day per week operation.

ACTIVITY	WASTE FLOW
GB Plant	3 gpm
Mustard DeMiliterization	1.5 gpm
Hydrazine Blending	3 gpm
Laundry	2 gpm
Quality Surety Lab.	1 gpm

TABLE 2

PESTICIDE PRODUCTION

SAMPLE DAY NO DATE DAY OF WEEK	1 2/9 Tue	2 2/10 Wed	3 2/11 Thur	4 2/12 Fri	5 2/13 Sat	6 2/14 Sun	7 2/15 Mon	8 2/16 Tue	9 2/17 Wed	10 2/18 Thur	11 2/19 Fri	12 2/20 Sat
Product												
Aldrin	1	1	1	2	1	1	1	1	1	1	2	1
Dieldrin	2	-	-	-	-	-	-	-	-	-	-	-
Endrin	2	2	-	-	-	-	-	-	-	-	-	-
Nemagon C	2	2	2	2	1	-	-	-	-	-	-	-
Dibrom	1	1	3	3	1	-	1	1	3	2	3	1
Vapona	2	5	4	5	2	-	1	4	4	5	4	-
Parathion	6	6	6	7	5	6	6	6	6	5	6	6

b. Industrial Waste Flow Rates.

(1) Flow into Lake F-1.

(a) Figure 3 presents graphically the two-hour average flows recorded at sampling point 1 for the twelve sampling days. A complete listing of flow data for this plot is tabulated in Appendix II.

(b) Table 3 lists the two-hour maximum, minimum, and daily average flow into Lake F-1. The overall average, 266.6 gpm, results in a total waste flow of 4.61 million gallons during the twelve-day survey. This is a substantial increase in flow over the value found in the 1960 USAEHA survey of 131 gpm average for the period 9-22 June 1960.

TABLE 3

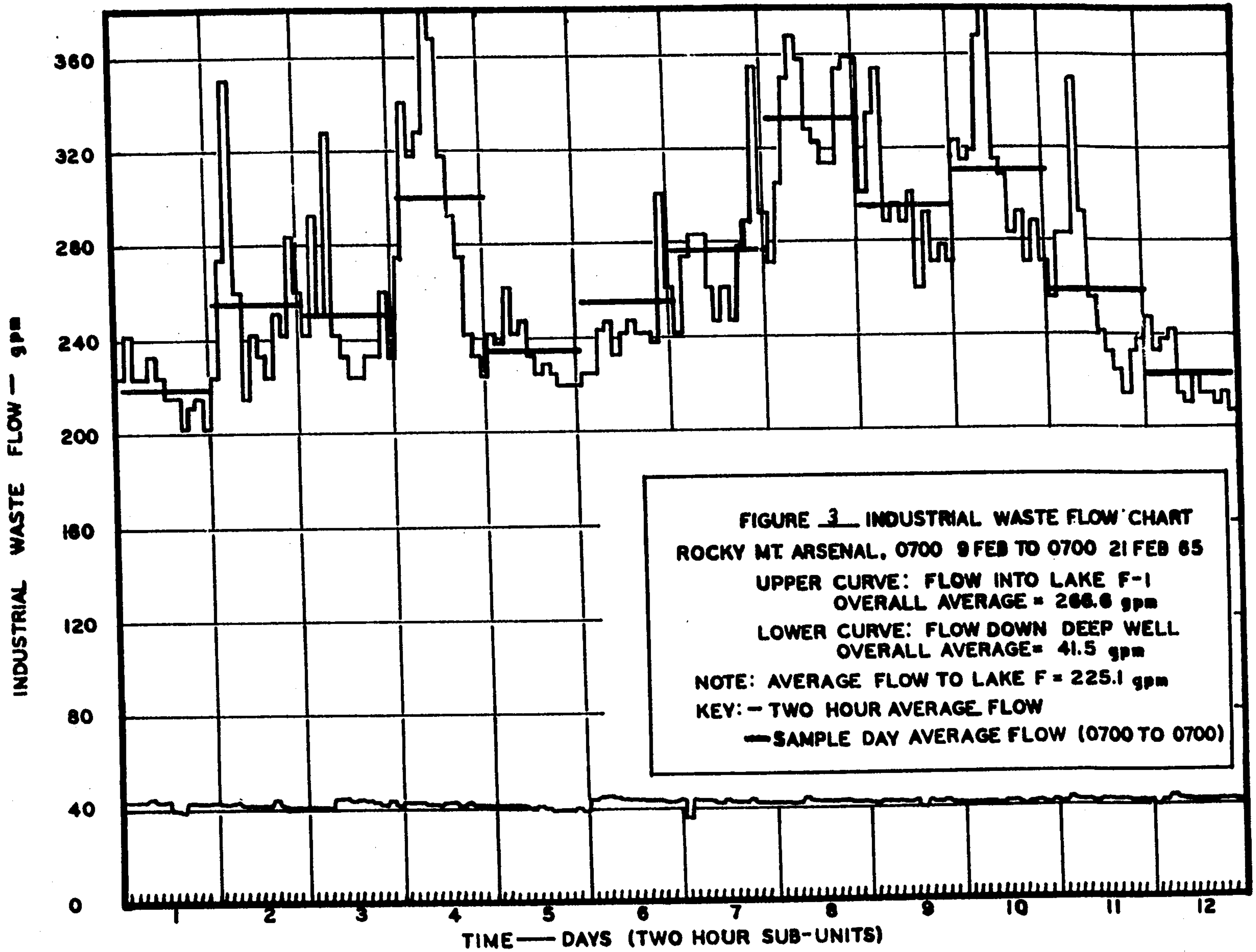
AVERAGE FLOWS INTO LAKE F-1

Sample Day No.	Day of Week	Min 2 hr-Avg (gpm)	Max 2 hr-Avg (gpm)	Daily Avg (gpm)
1	Tuesday	202*	242	219
2	Wednesday	215	350	255
3	Thursday	224	328	250
4	Friday	233	382	300
5	Saturday	220	261	234
6	Sunday	225	301	245
7	Monday	242	354	276
8	Tuesday	271	368	333
9	Wednesday	260	354	295
10	Thursday	271	386**	301
11	Friday	215	350	259
12	Saturday	207	247	223
			AVERAGE DAILY AVERAGE	266.6

*Minimum 2 hour-flow recorded during survey
**Maximum 2 hour-flow recorded during survey

(2) Rate of Injection into Deep Well.

(a) A plot of the two hour average flow is shown in Figure 4. Changes in filter operating conditions are also indicated in Figure 4 at the appropriate times. One filter was on line at a time for


INDUSTRIAL WASTE FLOW — gpm
360
320
280
240
200
160
120
80
40
0
1
2
3
4
5
6
7
8
9
10
11
12
TIME — DAYS (TWO HOUR SUB-UNITS)
FIGURE 3 INDUSTRIAL WASTE FLOW CHART
ROCKY MT. ARSENAL, 0700 9 FEB TO 0700 21 FEB 65
UPPER CURVE: FLOW INTO LAKE F-1
OVERALL AVERAGE = 266.6 gpm
LOWER CURVE: FLOW DOWN DEEP WELL
OVERALL AVERAGE = 41.5 gpm
NOTE: AVERAGE FLOW TO LAKE F = 225.1 gpm
KEY: — TWO HOUR AVERAGE FLOW
— SAMPLE DAY AVERAGE FLOW (0700 TO 0700)

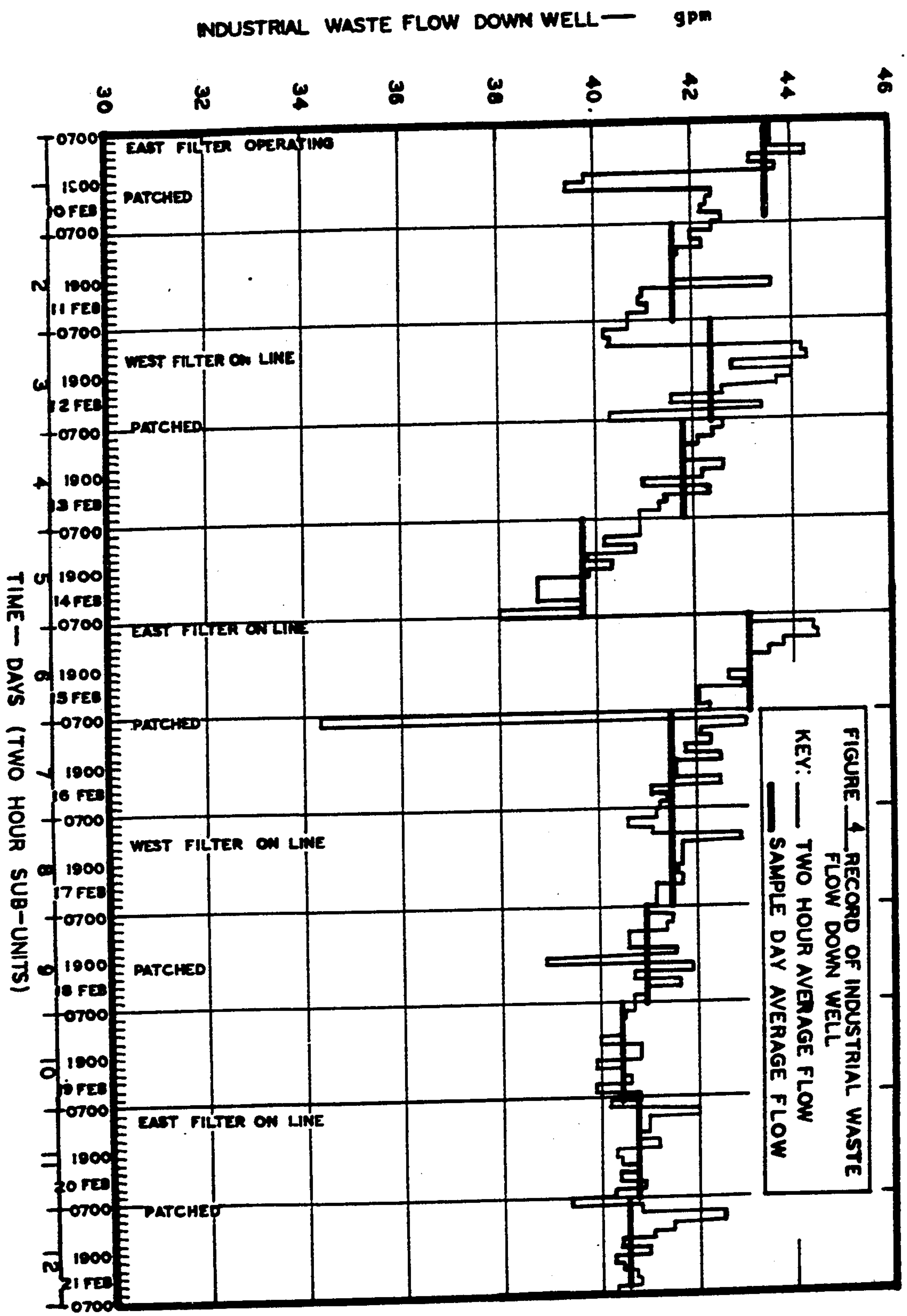


FIGURE 4 RECORD OF INDUSTRIAL WASTE FLOW DOWN WELL
KEY: TWO HOUR AVERAGE FLOW
SAMPLE DAY AVERAGE FLOW
INDUSTRIAL WASTE FLOW DOWN WELL — gpm
30
32
34
36
38
40
42
44
46
EAST FILTER OPERATING
PATCHED
WEST FILTER ON LINE
PATCHED
EAST FILTER ON LINE
PATCHED
WEST FILTER ON LINE
PATCHED
EAST FILTER ON LINE
PATCHED
10 FEB
11 FEB
12 FEB
13 FEB
14 FEB
15 FEB
16 FEB
17 FEB
18 FEB
19 FEB
20 FEB
21 FEB
0700
1900
TIME — DAYS (TWO HOUR SUB-UNITS)

runs of approximately 2.5 days. During each run, filter cakes were patched when analysis indicated leaks. The down time for switching or patching filters corresponds to the lower average injection rates.

(b) Table 4 lists the 2 hour maximum, minimum and average daily flow down the deep well. The overall average rate of 41.5 gpm indicates 0.717 million gallons of industrial waste were injected down the deep well for the twelve day survey period. This is 15.5 percent of the amount of industrial waste produced during the period.

TABLE 4

AVERAGE FLOWS DOWN INJECTION WELL

Sample Day No.	Min 2 hr-Avg (gpm)	Max 2 hr-Avg (gpm)	Daily Avg (gpm)
1	39.4	44.3	43.15
2	40.7	43.6	41.64
3	40.3	44.4	42.38
4	40.9	42.6	41.83
5	38.0	40.9	39.67
6	42.1	44.5*	43.12
7	34.3**	43.0	41.49
8	40.6	42.9	41.50
9	38.9	41.9	40.94
10	39.9	40.8	40.40
11	40.2	42.0	40.75
12	39.4	42.5	40.55
		AVERAGE DAILY AVG	41.5

*Maximum 2 hour flow recorded during survey
**Minimum 2 hour flow recorded during survey

c. Standard (On Site) Chemical Analyses.

(1) Two Hour Grab Samples. Figure 5 shows plots of the results of the analyses of grab samples from sample point 1. Waste properties that were measured are: Temperature, pH, turbidity, alkalinity and conductivity. The conductivity sensing electrodes became inoperative on sample day 9, terminating the conductivity results. Figure 6 graphically presents the results of analyses of grab samples from sample points 2 and 3. The analyses reported are the same as those in the previous figure.

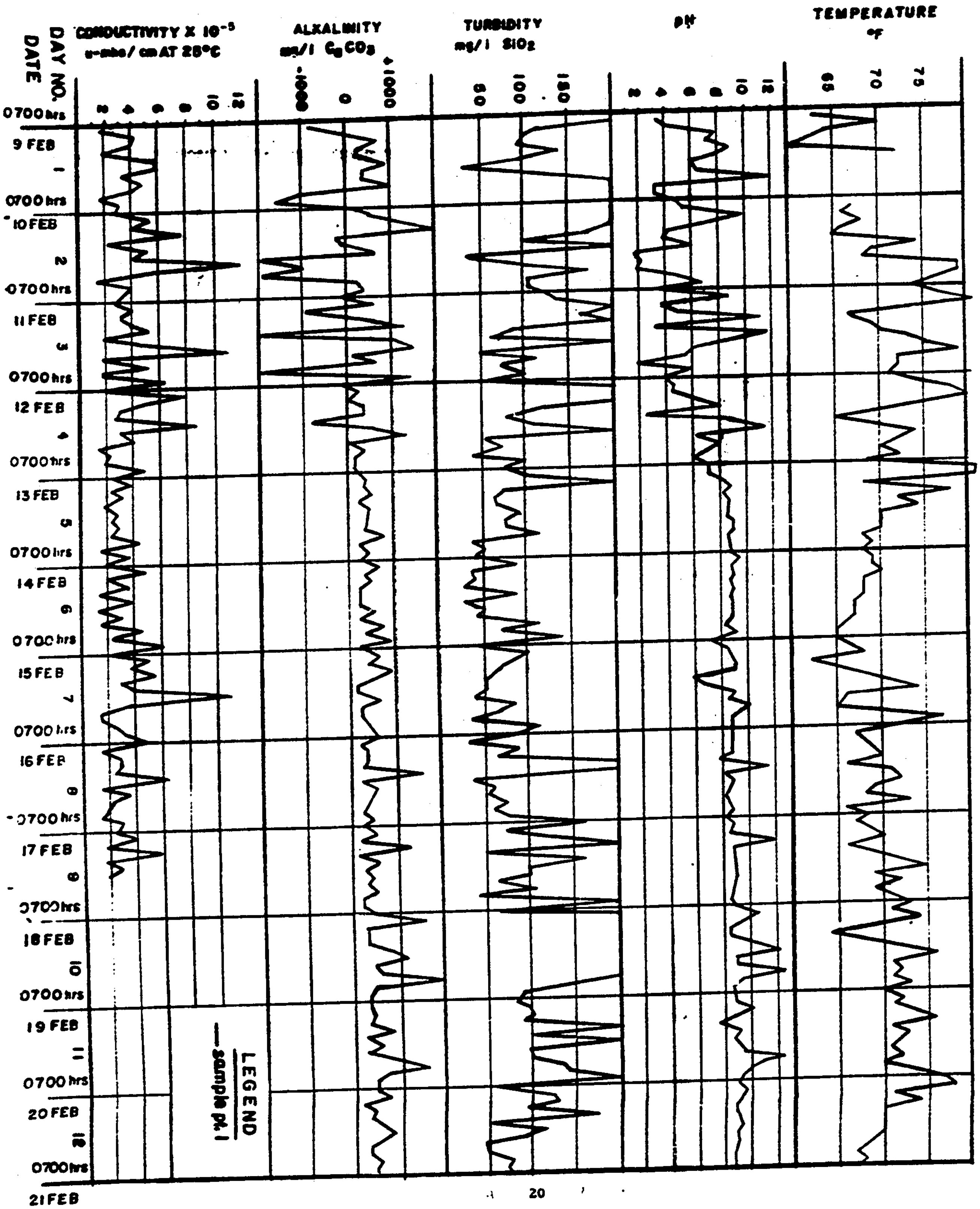


FIG. 5 - GRAB SAMPLE DATA FROM POINT I

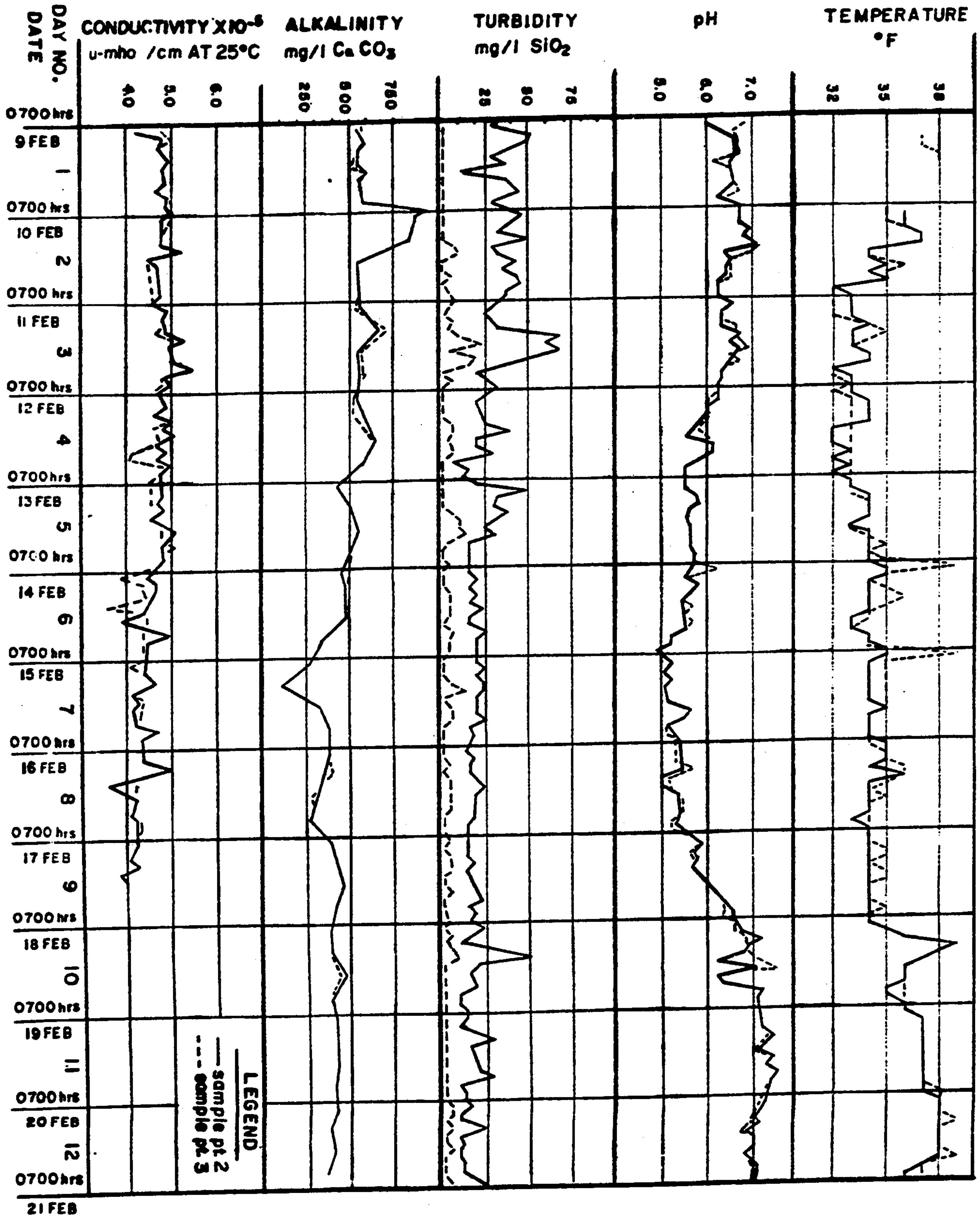


FIG. 6 GRAB SAMPLE DATA FROM POINTS 2 AND 3

(2) Daily Composite Results. A summary of the analyses performed on the composite samples is presented in Table 5. The average, standard deviation, maximum and minimum of the twelve day sampling period are tabulated for all three sampling points. Complete analytical data for composite samples is presented in Appendix II.

(3) Precision Tests. A test of the reproducibility of analytical methods was performed on eight of the standard chemical determinations. The results are presented in Table 6. Eleven consecutive analyses were performed on the same sample by each method. The laboratory work was done in the USAEHA laboratory at Edgewood Arsenal and completed on 16 April 1965.

d. Special Analyses.

(1) Pesticides. A summary of the results of pesticide analyses are shown in Table 7. The average, minimum and maximum for each sample point and analysis are indicated. From sample point 1, ten of the composite samples were tested and from sample points 2 and 3, only three composite samples were tested (sample days 4, 7 and 11) to make up the averages. As indicated, no detectable amounts of dibrom and nemagon C were found in the samples. Complete data on individual pesticide analyses results are shown in Appendix II.

(2) Also included in Table 7 are the results of a grab sample taken from Lake F at the end of the pumphouse pier. Although they are the results from a single determination, they indicate significant pesticide concentrations in the liquid of Lake F.

(3) Solid Samples. Bottom deposit samples were taken at two locations, sample points A and B (as indicated in Figure 7), and analyzed for pesticide concentrations. The results are shown in Table 8 and given as grams of pesticide per liter volume of mud. Also results are shown of the analysis of the still bottoms and production filter cake waste which the Shell Company dumps into Lake F along the east bank.

(4) General Observations.

(a) Visual observation of the flow patterns of the congo red dye used by the laundry indicated a considerable amount of short circuiting from the inlet of Lake F-1 to the overflow into Lake F, as indicated in Figure 7. Flow to the treatment building inlet proceeded across sample point A (Figure 7) directly along the north side of the Lake F-1. At sample point B (Figure 7), the lake appeared stagnant. These observations were qualitatively confirmed by observation of rate of

TABLE 5

SUMMARY OF STANDARD CHEMICAL ANALYSES OF DAILY COMPOSITES

Analyses (units)	1960 Survey Avg	SAMPLE POINT 1 Avg X	SP 1 Range of Values Min	SP 1 Range of Values Max	SP 1 Std Dev	SAMPLE POINT 2 Avg X	SP 2 Range of Values Min	SP 2 Range of Values Max	SP 2 Std Dev	SAMPLE POIN[T 3] Avg X	SP 3 Range of Values Min	SP 3 Range of Values Max
Acidity (mg/1)			-161	1057			42	235			47	266
Alkalinity (mg/1)			158	676			258	614			247	608
Arsenic (mg/1)	40.0	41.3	26.0	85.0	16.5	32.4	15.0	54.0	21.6	31.1	16.0	47.0
COD (mg/1)	194,000	2295	701	4080	1305	2196	1650	2928	330	2078	1670	2460
Chlorides (mg/1)	2710	681	518	798	95.8	835	686	918	85.1	848	696	954
Fluorides (mg/1)	2.14	9.2	<0.1	40.0	11.2	11.3	8.9	13.2	1.3	11.3	8.8	14.0
Iron (mg/1)	8.96	1.18	0.27	2.62	0.87	0.38	0.18	0.62	0.12	0.21	0.10	0.51
Nitrogen, Ammonia (mg/1)		2.1	<0.2	11.7	4.4	1.75	0.56	3.22	0.79	2.31	1.68	4.50
Nitrogen, Kjeldahl (mg/1)		0.60	0.28	0.84	0.16	0.55	0.14	1.12	0.30	0.60	0.28	1.12
Phosphorus Total, (mg/1)	45.6	183	25	710	201	261	130	410	95.5	235	100	413
Phenols (mg/1)	1230	719	54	3910	1050	557	272	1657	364	530	272	1657
pH (units)	7.18	7.4	3.9	10.5	2.4	6.2	5.0	7.5	0.88	6.2	5.0	7.5
Spec Conductance (μ-mhos/cm)	9.97×10^3		2.54×10^5	4.33×10^5			3.72×10^5	4.98×10^5			3.72×10^5	4.85×10^5
Sulfates (mg/1)	200.0	362	215	642	148	460	377	581	61	444	389	500
Sulfides (mg/1)		2.6	0.5	6.0	2.0	4.6	2.0	9.0	2.2	3.8	2.0	9.0
Solids, Dis (mg/1)	11,195	2631	1866	4105	780	3709	2695	4272	565	3723	2677	4231
Solids, Set (mg/1)	10.0	0.45	0.03	3.25	1.00	<0.01		<0.01		<0.01		<0.01
Solids, Sus (mg/1)	33.2	158	40	334	106	8.9	5.6	19.0	3.9	3.3	1.0	10
Solids, Volatile Sus (mg/1)		73.5	17.0	149	48.1	4.4	3.2	11.0	2.5	1.13	0.2	2.4
Solids, Tot Vol (mg/1)	5130	815	330	1560	378	1284	480	2000	517	1332	46[illegible]	2910
Solids, Tot (mg/1)	11,400	2784	2010	4170	735	3716	2700	4280	567	3726	2[illegible]	4241
Turbidity (mg/1)	494	131	64	>200	65	19.7	8.0	38.0	9.8	3.8	<1.0	8.0
Calcium (mg/1)		~300				~300				~300		
Magnesium (mg/1)		132.1	29	243		131.1	98	170		192.4	154	238
Aluminum (mg/1)		269.4	143	438		254.9	142	342		320.9	251	367

TABLE 6

PRECISION TEST

ANALYSIS	Results of Consecutive Analyses on Same Sample (Mg/l) (Sample Point 2, 0900, 22 Feb 65)											Average X	Standard Deviation
Chlorides	695	695	690	695	695	699	699	699	690	695	695	695.2	3.1
COD	1254	1122	1132	1141	944	972	1047	1245	1235	1273	1160	1139.0	112.6
Fluoride	11.6	11.6	11.6	9.2	9.0	9.0	10.2	10.2	9.6	10.8	11.2	10.36	1.06
Iron	0.36	0.30	0.29	0.27	0.30	0.29	0.27	0.30	0.32	0.30	0.28	0.298	0.025
Nitrogen, Ammonia	0.70	0.84	0.98	0.84	0.98	0.84	0.98	0.98	0.84	0.98	0.98	0.904	0.096
Nitrogen, Kjeldahl	1.12	1.26	1.12	1.26	1.26	1.12	0.98	1.12	0.98	1.12	1.26	1.145	0.105
Sulfates	410	410	450	475	410	410	475	500	450	475	410	443.2	34.4
Sulfides	1.5	1.3	1.3	1.3	1.3	1.3	1.7	1.4	1.5	1.3	1.3	1.38	0.13

TABLE 7

SUMMARY OF PESTICIDE ANALYSIS RESULTS

PESTICIDES (Concentrations in µg/l)	SAMPLE POINT 1			SAMPLE POINT 2			SAMPLE POINT 3			Grab Sample from Lake F
	Avg	Min	Max	Avg	Min	Max	Avg	Min	Max	
Aldrin	42.0	13.1	75.6	21.3	16.6	24.5	21.5	8.7	43.1	42.1
Dieldrin	68.3	14.5	125.2	35.6	25.1	49.1	38.2	19.9	64.3	43.5
Endrin	93.3	26.1	200.0	59.3	42.7	78.8	52.5	35.2	80.0	53.8
Parathion	367.0	126.4	945.1	167.7	138.6	186.1	182.2	129.1	265.9	123.8
Methyl Parathion	73.9	48.0	94.9	93.7	77.9	103.7	77.5	63.4	100.8	<7.0
Dibrom	None Detected			None Detected			None Detected			ND
Nemagon C	None Detected			None Detected			None Detected			ND

LAKE F-1
FIGURE 7

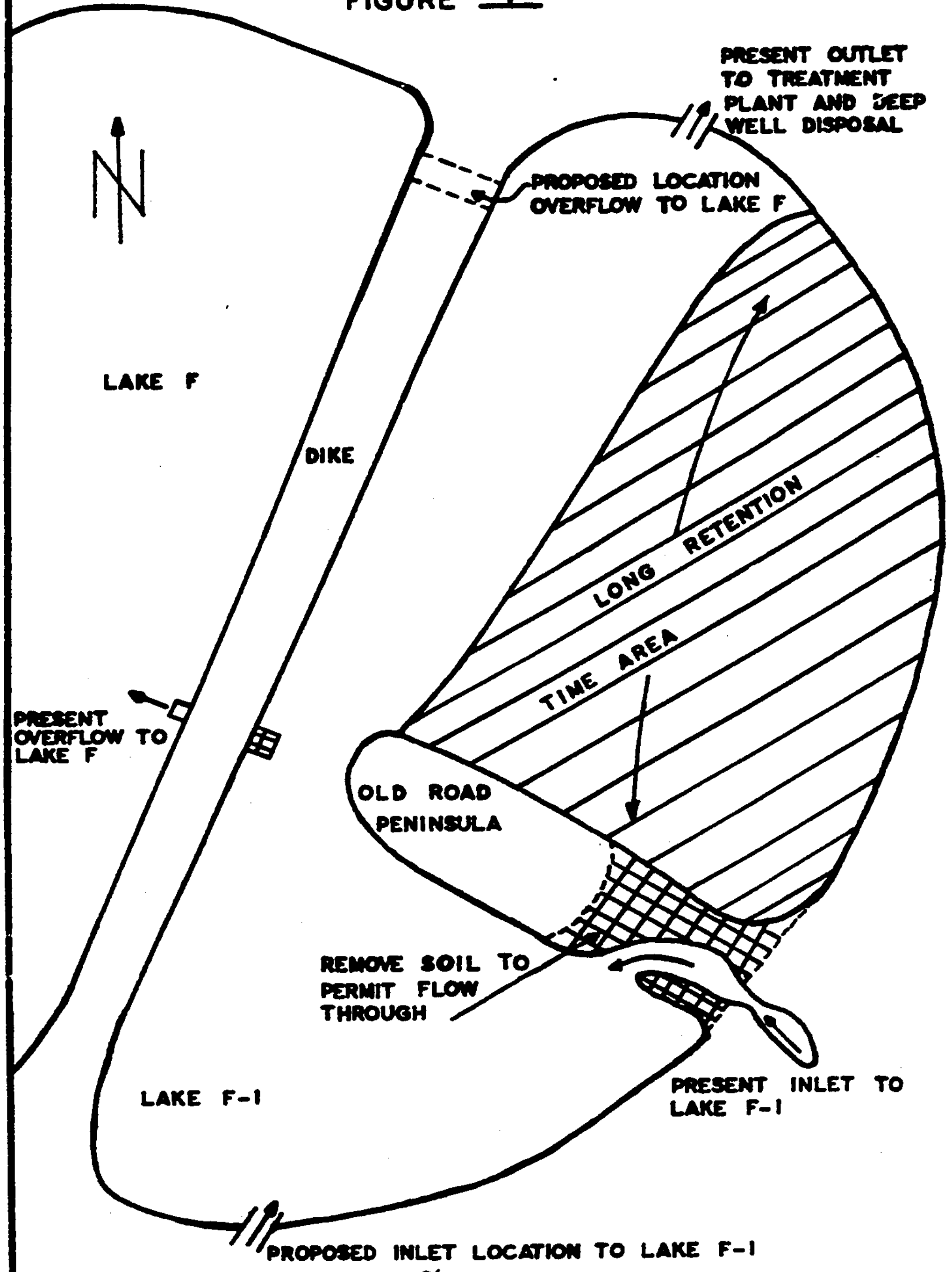


TABLE 8

SOLID SAMPLE PESTICIDE RESULTS

Pesticide Concentration (gm/l)	LAKE F-1 MUD SAMPLES Location A	Location B	Shell Process Still Bottoms
Aldrin	0.410	0.0217	0.324
Dieldrin	0.490	0.0771	0.210
Endrin	0.4129	0.0151	0.350
Parathion	1.104	0.0531	1.080
Methyl Parathion	0.204	0.0154	None Detectable

surface ice formation and melting. Approximate estimates of the Lake F-1 effective detention time were four days as compared to a calculated theoretical detention time of 9.8 days. These figures are based on the average flow into Lake F-1 and average overflow into Lake F.

(b) General observations were also made of the effect that surface storage of toxic industrial waste in Lakes F and F-1 has on the environment. Many sightings were made of the toxic effect on naturally occurring wild life. Along the banks of Lake F-1 and Lake F were found remains of dead ducks, rabbits, birds and mice.

7. DISCUSSION.

a. Production Activities.

(1) Shell Company Pesticide Production.

(a) Table 2, which lists the pesticide production by batches, indicates that three pesticide runs were terminated during the survey. Although the runs of individual pesticide may have been discontinued on a given day, residuals of the pesticide may continue to flow into the industrial waste stream for several more days because of washdown and clean-up procedures. The survey was conducted during the period 8-21 February 1965 because the large variety and amount of pesticide production scheduled during this period represented the maximum production activity of Shell for any two week period during the year. Table 2 also indicates that although Shell operates seven days a week, their production on weekends is greatly reduced.

(b) Over 90 percent of the water used, excluding water for power production, is consumed by Shell's industrial operations. All liquid production wastes except uncontaminated cooling water of Shell are discharged to the industrial waste disposal system.

(2) Other Rocky Mt. Ars. Industrial Waste-Producing Activities. Since physical conditions render it impossible to meter the flow from individual activities into the industrial waste disposal system, only estimates can be made of these flows. The GB plant was not producing agent during the survey and its only activities were filling projectiles and some agent redistillation. The estimated flow reported was provided by the operating staff at the GB plant. The mustard demiling activity was operating, but there was no waste flow since the shells were air cooled. Other flow estimates listed are approximate but indicate a level of magnitude.

b. Industrial Waste Flow Rates.

(1) Flow into Lake F-1.

(a) The two-hour average flows illustrated in Figure 3 point out considerable variation indicating many "slug" discharges. Within a single day as much as 60 percent variation in flow was observed with the highest flows occurring during the daylight hours of week days. On sample days 4 and 10, the peak discharges, occurring between 1500 and 1700 hours, appear to be related to high flows from the GB plant.

(b) The total industrial waste flow rate was over two times that found in the 1960 survey by this Agency. This increase is primarily due to Shell's increased production, alterations in the cooling system and the discharge of condenser jets to the industrial waste sewer.

(c) The Parshall Flume used to measure the flows produced reliable results. The manufacturer's specification state an accuracy of 1 ± 3 percent. The continuous depth recorder was manually recalibrated every twenty-four hours.

(2) Rate of Injection down Deep Well. The variations in the expanded plot of average injection flows shown in Figure 4 correspond directly to alterations in filter operation. Whenever there was down time for filter repair or change-over, the average flow was reduced. Also Figure 4 shows that for a given filter, the longer the running time the lower the flow rate. A comparison between the flow down the deep well and the total industrial waste flow is shown in Figure 3. With about 85 percent of the waste flowing into Lake F, this clearly emphasizes that increased injection rates or decreased incoming industrial flows are necessary if the accumulation in Lake F is to be stopped.

c. Standard Chemical Analyses.

(1) Two-hour Grab Samples and Composites.

(a) In general, the physical and chemical analytical results of the inlet stream into Lake F-1 shown in Figure 5 showed definite stabilization on weekends. The increased variation during week days emphasizes the batch nature of the discharges and the increased production activity. Figure 5 shows the pH as significantly stabilizing on sample day 5. The periodic increases of pH on days 7 through 11 correspond to periods of high flow shown on Figure 3. It can be noted that the alkalinity very closely follows the pH.

(b) The dampening effects of Lake F-1 on industrial waste characteristics can clearly be seen by comparing the results from sample point 1 in Figure 5 with those from sample points 2 and 3 shown in Figure 6. Since the only difference between sample points 2 and 3 is passage through a diatomaceous filter, all properties shown in Figure 6 other than turbidity closely follow one another. Differences in temperature reflect filter alterations and changes. The pH levels indicate about a five day lag between sample points 1 and 2. Average turbidity removal across the filter was about 25 mg/l and all of the waste going down the deep well and a turbidity of $>$20 mg/l.

(2) Daily Composite Results.

(a) Acidity and Alkalinity. Only maxima and minima are reported in Table 5 because of the characteristics of these analyses. Results from sample point 1 are quite variable, but those from sample points 2 and 3 are more uniform.

(b) Arsenic. Results were reasonably consistent and similar at all three sample points. The values are in the range of the background level in the water supply system. At sample point 1 the results agree closely with the findings in this Agency's 1960 survey.

(c) Chemical Oxygen Demand (COD). From Table 5 the lower standard deviations of COD results from sample points 2 and 3 clearly indicate the stabilizing effect of Lake F-1. Data from sample point 1 showed significantly lower values on weekends when compared with week day figures. Comparison of this data with the results from the 1960 USAEHA survey show the present waste to contain a much lower concentration of organics.

(d) Chlorides. Chloride values from sample points 2 and 3 were higher than those from sample point 1 but they showed a general downward trend during the survey. This could be accounted for if there had been previous high concentrations of chlorides in the waste stream just before the survey started. Again, comparison between chloride results of this and the previous survey indicate that the waste stream was about four times as concentrated in 1960.

(e) Fluorides. Although the average results from all three sample points were similar, the tenfold decrease in magnitude of the standard deviations for sample points 2 and 3 indicate a much greater consistency in values at these points. The maximum value at sample point 1 of 40 mg/l on the tenth day appear to be related to a slug of waste pumped from the GB plant sump. These results indicate that there is much more fluoride in the waste stream than when the 1960 survey was performed. This can be attributed to agent redistillation during the survey.

(f) Iron. The values of iron concentration found at sample point 1 were much higher than those at sample points 2 and 3. This removal could occur by precipitation or sedimentation. Also the inlet waste iron values were much lower on weekends. The iron concentration in the 1960 survey was about seven times higher.

(g) Ammonia Nitrogen. The procedure used in this determination was quite variable and appeared to measure not only ammonia but also all volatile organic bases present. The very high values at sample point 1 occurred on the last two days of the survey.

(h) Kjeldahl Nitrogen. Within the variability of the analytical method used all three sample points showed similar results. Data show a definite upward trend in the latter half of the survey.

(i) Total Phosphorus. Average results for sample point 1 were substantially lower than those at sample points 2 and 3. The latter two sample points produced results showing a definite downward trend which would account for this difference. Sample point 1 showed greatly reduced concentrations on weekends. The average level found in the 1960 survey was much lower than the current survey. This can be accounted for by increased production of organic phosphorus pesticides.

(j) Phenols. Concentrations found at sample point 1 were much more variable than those at sample points 2 and 3, which were very similar. All positions exhibited a slight downward trend after sample day 4. Again sample point 1 results showed a definite decrease during weekends. The 1960 survey phenol results were just slightly higher than those discussed above.

(k) pH. The pH results from the daily composites are very similar to those reported earlier for the two-hour grab samples. The data clearly show the dampening effect of Lake F-1 and that sample points 2 and 3 exhibit a lag time of about four days behind changes at sample point 1. The average pH of the waste in 1960 was very close to that found during this survey.

(l) Specific Conductance. Only a limited amount of data were obtained because the instrument became inoperative during the survey. However, the values obtained were much higher than those found in 1960.

(m) Sulfates. As seen by a comparison of the standard deviations among the three sample points on Table 5, the dampening effect of the system is clearly observed. Values for sample point 1 were much

lower on weekends. Also, the levels of sulfates are about double that found in the 1960 survey. Vapona production can be related to the increase in sulfates.

(n) Sulfides. The method used to measure sulfides was not very precise. However, results indicate a significant increase across Lake F-1. This may be due to the presence of some sulfide producing action.

(o) Solids. Results presented in Table 5 indicate that most of the solids present in the waste are in dissolved form. The total solids appeared to increase across Lake F-1 while the suspended solids were greatly reduced by sedimentation. Because of the cold weather and ice cover, little evaporation occurred to concentrate the dissolved solids. The solids results from the 1960 survey indicate that at the time the waste was about five times more concentrated than it was during this survey. Because there are many volatile organics present in this waste, it is important to point out that solid results will not include these substances.

(p) Turbidity. These results are similar to those from the two hour grab samples and show the effectiveness of sedimentation in Lake F-1 and pressure filtration in producing an effluent of an average turbidity of about 4 mg/l for injection down the deep well. Strong winds increase the turbidity level at sample point 2.

(q) Calcium, Magnesium and Aluminum. Flame spectrophotometry showed the calcium content in the daily composite to run about 300 mg/l at all sample points so this value was used as an internal standard for magnesium and aluminum determinations. Average results from sample points 1 and 2 for magnesium and aluminum are very similar and establish the concentration levels present. The reason for the higher values reported for sample point 3 is related to the use of calcium as an internal standard and its obvious increase across a diatomaceous filter.

(3) Precision Tests. The precision tests of some of the standard chemical analytical methods are shown in Table 6. A quantity of waste from sample point 2 was used for these tests but it was five weeks after sample collection before these tests were completed. Significant visual change had taken place in the sample during that time. Due to the complex nature of this waste and interferences present, more variability was found than anticipated. However, these results do give an indication of the reproducibility of the analytical methods employed.

d. Special Analyses.

(1) Pesticides

(a) Aldrin. Results from sample points 2 and 3 agree quite closely and were about half the average value found at sample point 1. Although the production schedule of aldrin was quite constant throughout the survey, the concentrations in the inlet stream at sample point 1 were greatly reduced on weekends.

(b) Dieldrin. The production of this pesticide was stopped the second day of the survey, at which time the highest concentration was found in the inlet waste stream at sample point 1. However, lower peak inlet concentrations were also observed on sample days 4 and 8 with the weekend showing very reduced values. Results from sample points 2 and 3 exhibited much less variability, had an average of about half that at the inlet and showed about a four day lag time of the effect of peaks at sample point 1.

(c) Endrin. Similar to the other two chlorinated pesticides, Lake F-1 was shown to have about a 50 percent attenuation effect on inlet concentrations. The production of endrin was terminated on sample day 2. There was a general downward trend of inlet results after that time, but similar to dieldrin, there was a definite peak on sample day 8. This may have been caused by washing activities on that day. The closeness of the average results from sample points 2 and 3 indicate the free passage of the pesticides through the filter and the approximate precision of the analytical methods used.

(d) Parathion. The highest pesticide concentrations found were those of parathion ranging up to 1000 µg/l. Although the production was consistent throughout the survey (as shown in Table 2), the inlet concentration showed distinct peaks on sample days 3, 7 and 11. These periodic discharges are probably due to the Shell's policy of accumulating and hydrolyzing the parathion waste before discharging it into the industrial waste stream. Results from sample points 2 and 3 were much less variable and about half the average inlet concentrations. The differences in averages between sample points 2 and 3 are felt to be insignificant considering the number of samples and analytical procedures.

(e) Methyl Parathion. Results of analyses for this pesticide were fairly similar for all sample positions and sample times. Since no methyl parathion was produced during the survey period, the concentrations are either due to residuals from previous runs or from certain breakdown products of parathion. The data did not reflect any of the variations shown by the parathion results.

(f) Other Pesticides. The particular analytical methods used showed no detectable levels of dibrom or nemagon C present. More

sensitive procedures might establish that low levels of these pesticides are present; however, considering the nature of production by Shell, no significant concentrations of these pesticides are expected in the waste stream.

(2) Solid Samples. The results in Table 8 indicate very high pesticide concentrations in solid samples taken from the bottom of Lake F-1. Values from the sample taken in the direct line of flow (Location A, Figure 1) are very high. The lower levels at Location B confirm the visual observation that there is a somewhat stagnant zone in that portion of the lake. Most of the pesticide concentrations were reduced about 50 percent between the inlet and outlet of Lake F-1. The pesticides apparently settle out accounting for the high concentration in the bottom material.

(3) General Observations. Chemical results have shown that Lake F-1 does have a significant dampening effect to variations in the inlet waste stream; however, greater buffering effect could be achieved if the stagnant areas were eliminated. This could be done by relocation of the inlet, the overflow to Lake F, and the removal of the center dike. Attaining the maximimum retention will become more important when the overflow to Lake F is eliminated. If there were no flow into Lake F, the retention time in Lake F-1 for the 266 gpm average flow would be approximately 2.6 days.

8. CONCLUSIONS.

a. A high volume of waste is still being contributed to Lake F and evaporation rates will not keep up with the rate of excess waste flow.

b. Chemical and physical properties of the wastes at the inlet of Lake F-1 were highly variable. Lake F-1 does exert some dampening effect on the variability of the waste properties; however, increased detention time would be desirable.

c. Wastes analyzed during this survey were more dilute than those analyzed during the 1960 survey conducted by this Agency. The addition of slightly contaminated cooling water to the industrial waste system accounts for the increased volume and more dilute nature of the waste. The greater volume means total cost of disposal has increased.

d. Industrial wastes from still and process tank bottoms, which are trucked out and emptied in Lake F, are high in pesticide content.

e. The toxic effects on the environment of surface storage of this industrial waste in Lake F-1 and Lake F are quite obvious, particularly on wild life.

f. There were high pesticide concentrations in Lake F-1 liquid and a significant build-up in bottom deposits. Lake F also contained significant pesticide concentrations.

9. RECOMMENDATIONS.

a. Dispose of all currently generated industrial wastes through the deep well disposal system and eliminate the build-up of waste volume in Lake F.

b. Change the overflow and inlet location of Lake F-1 and modify the peninsula in Lake F-1 to attain greater settling and equalizing effects. The new inlet location can be achieved by surface diversion or surface piping.

c. Investigate waste quantity reduction steps and alternate disposal methods for the industrial wastes currently being produced.

d. Discontinue the current policy of disposal of still and process tank bottom wastes into Lake F or Lake F-1. Off waste Cinc? Fill? empty?

e. Take steps to eliminate Lake F, as soon as possible, and thereby remove much of the present environmental hazard of exposed surface storage of toxic wastes.

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APPENDIX I

SPECIAL LABORATORY PROCEDURES

1. Iron Determination.

a. Because of the large amounts of interfering compounds expected in the waste, Standard Methods[1] called for the use of the ether extraction technique. However, because of the large number of samples anticipated, a shorter, more simple technique was sought. Therefore, an investigation of modifications of the 2,2'2"-tripyridine technique[1] was performed in the SED laboratory utilizing known concentrations of expected interferences[2].

b. Through an investigation of the procedural variables, the pH of the system prior to color development was found to be very critical. As stated by both Standard Methods[1] and the original work[3], the volume of ethylenediamine to be used is 5 ml. This volume of base raised to pH to between 10.5 and 11, although the references stated that the pH should be 9.6. It was found that careful addition of ethylenediamine to give a pH of 9.6 eliminated the error due to phosphate. Other interfering metals were added to water and found to produce negligible error when the pH was properly adjusted. Experiments with samples of the waste indicated that residual nitric acid was affecting color development and therefore had to be removed by boiling the sample to dryness. Finally the standard addition test was performed using samples of the waste and gave acceptable results.

c. The procedure used was as follows:

(1) Use 50 ml of sample, or a known value, containing less than 0.2 mg iron, dilute to 50 ml.

(2) Add 3 ml HNO_3 and boil down to dryness.

(3) Add 20 ml water and 2 ml HCl.

(4) Heat and reduce volume by 30 percent.

(5) Cool.

(6) Add 1 ml Hydroxylamine-HCl.

(7) Adjust pH to 9.6 using Ethylenediamine.

(8) Add 5 ml Tripyridine and dilute to 100 ml.

(9) Measure optical density at 555 mu.

2. Arsenic Determination.

a. A determination with silver diethyldithiocarbamate was used which was adopted from the US Public Health Service, Robert A. Taft Sanitary Engineering Center recommendations[4]. The sensitivity and relative resistance to interferences made this determination favorable for the waste involved. A Gutzeit apparatus is used. The arsenic is reduced to the trivalent form, evolved as arsine and passed through a lead acetate scrubber into an absorber containing the silver diethyldithiocarbamate where the arsine forms a soluble red complex.

b. The procedure used was as follows:

(1) Use 50 ml of sample, or a known volume, containing less than 15 ug As, diluted to 50 ml.

(2) Add 5 ml of concentrated HCl, 2 ml of 15 percent KI solution and 8 drops of stannous chloride solution.

(3) Mix well and allow to stand 15 minutes.

(4) Impregnate the scrubber with lead acetate solution and place 4 ml of silver diethyldithiocarbamate solution in the absorber tube.

(5) Add 3 grams of granular zinc to the solution in the generator flask and connect the apparatus immediately.

(6) Let stand 30 minutes until arsine evolution is complete.

(7) Transfer the absorbing solution to 1 cm cells and measure absorbance at 560 mu in a spectrophotometer.

3. Total Phosphorous Determination.

a. For the determination of total phosphorous the procedures in Standard Methods[1] were inadequate because of the organic nature of the waste. A literature search disclosed two techniques[5,6] which appeared applicable for the anticipated waste. After preliminary laboratory analyses of organic phosphate ester compounds utilizing these two procedures, it was found that neither procedure yielded acceptable results, but that a modification of each combined into a third procedure[7] described below proved to be satisfactory.

b. The sample is brought to a pH of 8-10 in order to hydrolize the organic phsophate esters. It is then digested over low heat with ammonium persulfate to convert the phosphorous to orthophosphate. After

completion of the oxidation process (resulting in a colorless solution), the excess ammonium persulfate is destroyed by the addition of sodium sulfite. The sample is then neutralized with nitric acid using paranitrophenol as the indicator. Acid molybdate solution and aminonaphtholsulfonic acid are added for color development, the volume is adjusted, and an aliquot is placed in the spectrophotometer. Readings are made at 690 mu and compared against a standard curve prepared in the same manner.

4. Gas Chromatograph Analytical Specifications.

COLUMN: 5'5" of ¼" OD pyrex glass filled with a mechanical mixture of 1 part 5% DC-LSX-3-0295 on 60-80 mesh

Gas Chrom Z

1 part 20% SE-30 on 60-80 mesh

Gas Chrom P

Temperature - Isothermal at 209°C

DETECTOR: Electron Capture at 210°C

pulse internal - 1/150 Sec

pulse time - 0.75 u Sec

Injection Part Temperature: 225°C

Carrier Gas Flow: Argon-methane (95%-5%) - 85 ml/min

Purge Gas Flow: Argon-Methane (95%-5%) - 175 ml/min

Type of Instrument Used: F and M 810

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APPENDIX II

FLOW RATE DATA INTO LAKE F-1
(Sample Point 1)

	CONCENTRATION IN GALLONS PER MINUTE											
2 Hour Average	1	2	3	4	5	6	7	8	9	10	11	12
0700-0900	224	320	295	287	370	225	242	271	301	323	256	247
0900-1100	242	392	356	357	365	225	274	305	335	315	283	233
1100-1300	224	500	306	333	400	242	283	350	354	319	283	238
1300-1500	224	372	400	344	370	247	283	368	288	368	350	242
1500-1700	233	307	295	400	378	233	261	358	296	386	292	215
1700-1900	224	345	284	386	357	242	247	328	288	315	256	211
1900-2100	215	334	273	333	344	247	261	323	301	309	242	224
2100-2300	215	320	273	306	350	242	247	314	260	283	233	215
2300-0100	202	359	284	287	344	242	278	314	292	292	225	215
0100-0300	211	345	284	253	338	238	289	354	271	271	215	211
0300-0500	215	405	317	244	338	301	354	358	278	288	233	215
0500-0700	202	372	284	234	338	261	292	358	271	271	238	207
Daily Average	219	255	250	300	234	245	276	333	295	310	259	223

APPENDIX II (Cont)

FLOW RATE DATA DOWN DEEP WELL
(Sample Point 3)

2 Hour Average	CONCENTRATION IN GALLONS PER MINUTE 1	2	3	4	5	6	7	8	9	10	11	12
0700-0900	--	42.3	40.7	42.6	40.9	43.1	34.3	41.2	41.0	40.7	40.2	39.4
0900-1100	--	42.0	40.3	42.4	40.9	44.4	43.0	40.6	41.5	40.5	42.0	40.8
1100-1300	43.6	42.2	40.3	42.2	40.2	44.5	42.1	41.1	41.4	40.4	41.0	42.5
1300-1500	44.3	41.7	44.2	41.8	40.7	43.8	42.3	42.9	40.6	40.4	41.0	41.5
1500-1700	43.2	41.5	44.4	41.8	39.8	43.5	41.8	41.7	40.6	40.0	40.7	41.1
1700-1900	43.7	41.6	42.9	42.6	40.3	43.1	42.5	41.7	41.6	40.8	41.2	40.4
1900-2100	39.8	41.7	44.0	42.2	39.8	43.1	41.6	41.7	38.9	40.8	40.3	41.0
2100-2300	39.4	43.6	43.7	41.0	38.8	42.7	41.6	41.6	41.9	39.9	40.4	40.3
2300-0100	42.4	41.0	42.6	42.3	38.8	43.0	42.5	41.7	40.7	40.4	40.8	40.4
0100-0300	42.3	40.9	41.6	41.4	38.8	42.1	41.1	41.2	41.6	40.6	40.4	40.7
0300-0500	42.2	41.1	43.4	41.3	39.6	42.1	41.4	41.2	41.0	39.9	40.9	40.8
0500-0700	42.6	40.7	40.3	40.9	38.0	42.3	41.3	41.2	40.7	40.8	40.4	40.3
Daily Average	43.2	41.6	42.4	41.8	39.7	43.1	41.5	41.5	40.9	40.4	40.8	40.6

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APPENDIX II (Cont)

COMPOSITE RESULT DATA
(Sample Point 1)

Analysis	1	2	3	4	5	6	7	8	9	10	11	12
Acidity (mg/1)	1057	1024	889	484	11.2	10.3	34	0	-20.4	-161	-58	-41
Alkalinity (mg/1)	-158	-144	10	196	344	403	438	308	453	676	465	440
Arsenic (ug/1)	85	49	34	36	28	52	36	34	34	26	40	38
COD (mg/1)	4080	3890	3220	3120	840	701	1775	1460	4010	2230	1300	912
Chlorides (mg/1)	676	737	798	770	571	716	788	518	592	572	752	686
Fluorides (mg/1)	1.6	9.0	14.0	12.0	<0.02	4.0	0.3	13.5	12.7	40.0	2.5	0.4
Iron (mg/1)	1.10	2.62	1.72	2.10	0.76	0.42	2.55	1.30	0.27	0.47	0.25	0.30
Nitrogen Ammonia (mg/1)	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15	1.12	1.68	11.2	11.7
Nitrogen, Kjeldahl (mg/1)	0.84	0.56	0.56	0.56	0.42	0.28	0.84	0.70	0.56	0.70	0.56	0.56
Phosphates (mg/1)	75	710	240	230	52	70	<10	290	80	350	25	75
Phenols (ug/1)	3910	665	1090	798	245	162	263	517	377	445	97	54
pH	4.0	3.9	4.5	4.9	4.4	8.5	8.1	8.6	9.1	10.5	9.4	9.0
Spec. Conductance µ-mhos/cm x 10^{-5}	4.33	4.17	3.69	4.00	2.82	3.14	3.26	2.54	---	---	---	---
Sulfates (mg/1)	338	642	640	478	285	280	215	371	265	320	223	285
Sulfides (mg/1)	2.0	0.5	1.0	2.0	1.0	0.5	2.0	5.0	2.0	3.0	6.0	6.0
Solids, Dis. (mg/1)	3248	3289	4105	3382	1695	2130	2120	1663	1922	2906	2076	---
Solids, Settleable (mg/1)	0.025	0.065	0.035	0.030	0.030	0.035	0.045	0.060	0.050	3.25	1.25	[illegible]
Solids, Sus. (mg/1)	41	40	65	128	115	120	110	207	248	334	324	[illegible]
Solids, Vol. Sus. (mg/1)	22	17	30	100	---	54	54	97	113	149	140	32
Solids, Tot. Vol. (mg/1)	942	347	1560	1090	628	750	777	459	874	1210	330	---
Solids, Tot. (mg/1)	3249	3329	4170	3510	2010	2250	2230	2070	2170	3240	2400	---
Turbidity (mg/1)	240	208	68	64	96	72	76	140	84	>200	>200	120
Aluminum (mg/1)	---	143	296	206	---	164	387	259	263	438	274	264
Magnesium (mg/1)	---	105	29	76	---	74	142	171	140	116	225	243

APPENDIX II (Cont)

COMPOSITE RESULT DATA
(Sample Point 2)

Analysis	1	2	3	4	5	6	7	8	9	10	11	12
Acidity (mg/1)	70.6	61.4	74.4	143	196	235	210	192	116	60.5	42.0	50.5
Alkalinity (mg/1)	604	597	614	436	500	258	264	371	434	408	403	371
Arsenic (ug/1)	50	37	39	<15	54	48	50	42	36	<15	<15	34
COD (mg/1)	2928	2210	2240	2350	2450	2300	2300	2190	1990	1910	1840	1650
Chlorides (mg/1)	822	822	908	860	898	890	918	914	876	722	706	686
Fluorides (mg/1)	11.8	13.2	12.2	12.4	11.6	10.7	11.4	12.0	10.8	8.9	11.0	9.2
Iron (mg/1)	0.62	0.45	0.38	0.40	0.35	0.40	0.50	0.48	0.35	0.27	0.18	0.22
Nitrogen, Ammonia (mg/1)	0.56	1.40	3.22	2.80	2.52	1.82	1.12	1.26	1.12	1.40	1.40	2.38
Nitrogen, Kjeldahl (mg/1)	1.12	0.28	0.28	0.28	0.14	0.28	0.56	0.70	0.56	0.84	0.84	0.70
Phosphates (mg/1)	374	380	330	410	230	130	240	290	150	215	155	225
Phenols (ug/1)	556	272	1657	444	530	626	500	561	507	394	305	328
pH	6.7	6.7	6.5	5.5	5.6	5.0	5.0	5.4	6.1	7.1	7.5	7.2
Spec. Conductance μ-mhos/cm x 10^{-5}	4.98	4.67	4.09	4.79	4.68	445	3.72	4.20	---	---	---	---
Sulfates (mg/1)	473	422	517	473	493	581	427	523	427	395	377	410
Sulfides (mg/1)	5.0	5.0	8.0	3.0	4.0	2.0	4.0	9.0	6.0	2.0	4.0	3.0
Solids, Dissolved (mg/1)	4241	3839	4270	4272	4203	3812	3804	3236	3551	2862	2695	---
Solids, Settleable (mg/1)	<0.01											
Solids, Sus. (mg/1)	19.0	12.0	10.4	7.6	7.2	7.6	6.0	5.6	9.2	7.6	5.4	6.8
Solids, Vol. Sus. (mg/1)	11.0	4.0	7.2	2.4	3.6	2.8	3.2	3.6	3.2	4.4	3.2	4.0
Solids, Tot. Vol (mg/1)	1024	651	2000	1520	907	912	1930	1600	1390	1710	480	---
Solids, Total (mg/1)	4260	3851	4280	4280	4210	3820	3810	3240	3560	2870	2700	---
Turbidity (mg/1)	38	29	24	24	20	16	9	12	32	8	12	12
Aluminum (mg/1)	382	---	206	142	266	253	313	226	---	264	263	234
Magnesium (mg/1)	166	---	135	98	138	110	123	102	---	170	168	101

APPENDIX II (Cont)

COMPOSITE RESULT DATA
(Sample Point 3)

Analysis	1	2	3	4	5	6	7	8	9	10	11	12
Acidity (mg/1)	70.6	61.4	74.4	138	196	226	214	194	107	51.2	46.5	50.2
Alkalinity (mg/1)	608	604	592	468	479	268	247	372	430	425	398	360
Arsenic (ug/1)	47	31	24	32	32	20	44	32	40	24	16	34
COD (mg/1)	2110	2110	2210	2310	2460	1890	2260	2170	1990	1950	1800	1670
Chlorides (mg/1)	890	899	954	868	926	880	914	914	864	696	696	678
Fluorides (mg/1)	11.9	14.0	12.2	12.7	11.8	11.4	11.2	11.3	10.8	8.8	9.0	9.9
Iron (mg/1)	0.21	0.18	0.19	0.18	0.17	0.18	0.27	---	0.20	0.10	0.22	0.11
Nitrogen, Ammonia (mg/1)	4.50	1.96	2.94	2.94	2.52	1.68	1.82	1.82	1.68	1.68	1.68	2.52
Nitrogen, Kjeldahl (mg/1)	1.12	0.28	0.28	0.42	0.42	0.56	0.28	0.84	0.84	0.84	0.42	0.84
Phosphates (mg/1)	257	413	340	310	200	100	170	360	110	200	170	185
Phenols (ug/1)	349	272	1657	466	542	500	520	544	533	391	284	298
pH	6.7	6.7	6.5	5.6	5.6	5.0	5.0	5.4	6.1	7.2	7.5	7.2
Spec. Conductance µ-mhos/cm x 10^{-5}	4.85	4.62	4.26	4.62	4.56	4.33	3.72	4.20	---	---	---	---
Sulfates (mg/1)	453	435	500	427	460	493	378	467	491	414	389	415
Sulfides (mg/1)	3.0	6.0	4.0	3.0	2.0	2.0	3.0	9.0	5.0	2.0	5.0	2.0
Solids, Dis. (mg/1)	4231	4022	4227	4096	4208	3668	3718	3679	3537	2888	2677	---
Solids, Set. (mg/1)	<0.01											
Solids, Sus. (mg/1)	10.0	3.0	3.2	4.0	2.4	2.4	2.0	1.0	3.0	2.2	2.8	2.8
Solids, Vol. Sus. (mg/1)	<0.1	<0.1	2.0	2.4	1.6	---	<0.1	<0.1	0.8	1.6	1.6	2.4
Solids, Tot. Vol. (mg/1)	1035	708	1800	1895	2910	1470	955	749	1600	1070	460	---
Solids, Total	4241	4025	4230	4100	4210	3670	3720	3680	3540	2890	2680	---
Turbidity (mg/1)	8	1	8	7	<1	3	<1	2	8	2	2	2
Aluminum (mg/1)	319	327	367	367	320	468	306	326	251	---	---	260
Magnesium (mg/1)	148	212	205	193	216	213	175	154	170	---	---	238

APPENDIX II (cont)

COMPOSITE PESTICIDE RESULTS
(All concentrations in $\mu g/ml$)

Sample Day	Aldrin	Dieldrin	Endrin	Parathion	Methyl Parathion
Sample Pt. #1					
2	63.4	150.1	109.7	438	82.7
3	75.6	66.8	168.4	945	94.9
4	58.3	120.0	100.0	343	69.0
6	13.1	25.8	57.6	230	67.1
7	58.4	79.1	87.0	481	88.7
8	68.8	125.2	200.0	368	88.8
9	28.7	35.8	48.7	144	48.0
10	14.7	36.4	58.9	135	48.8
11	24.9	29.4	83.0	460	77.3
12	13.8	14.5	26.1	126	< 7.0
Sample Pt. #2					
4	24.5	33.2	56.3	139	99.8
7	22.7	49.1	78.8	179	103.7
11	16.6	25.1	42.7	186	77.9
Sample Pt. #3					
4	12.8	30.5	42.2	152	63.4
7	43.1	64.3	80.0	266	100.8
11	8.7	19.9	35.2	129	68.4